Effects of reducing the reactor diameter on the fluidization of a very dense powder

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A B S T R A C T

The impact of decreasing the column diameter from 5 to 2 cm has been studied on the fluidization of a very dense powder both at ambient and high temperature. The final objective was to coat very dense nuclear fuel particles with silicon using a Fluidized Bed Chemical Vapor Deposition (FB-CVD) process with bed weights as low as possible. A surrogate tungsten powder, 19300 kg/m^3 in density, was used. Wall effects only appeared in the column with a 2 cm diameter. This was evidenced by an increase in the hysteretic behavior of the pressure drop curves and an increase of the minimum fluidization velocity, as well as by a decrease in the bed voidage. This critical diameter is higher than those found elsewhere for Geldart's group A and B powders, which is probably due to the very high density of the tungsten powder that increases the friction forces in the bed. Satisfactory thermal conditions were reached at 650 °C in a reactor with a 3 cm diameter using only 740 g of powder, thereby opening the way for future silicon coating experiments.

Keywords:
Fluidized bed
Wall effects
Very dense particles
Minimum fluidization velocity
Pressure drop
Bed expansion

1. Introduction

New nuclear fuels with limited enrichment in ^{235}U are under development for research reactors. Pulverulent U(Mo) metallic fuels dispersed in an aluminum-based matrix are among the most promising materials today. The coating of U(Mo) powder by a barrier material is also being considered to limit interfacial interactions between the fuel and its matrix under irradiation [1]. Silicon coatings formed by Fluidized Bed Chemical Vapor Deposition (FB-CVD) from silane (SiH_4) is being investigated for the barrier [2].

For economic reasons, the FB-CVD process must be developed to treat fuel powder weights that are as low as possible. The fuel powder in question has a very high density (about 17500 kg/m^3) which is close to that of tungsten (19300 kg/m^3) [3]. This explains why a tungsten powder with mean diameter similar to that of the fuel powder has been used as a surrogate powder in previous studies [4]. Some preliminary experiments have demonstrated that the FB-CVD technology can uniformly coat 1500 g of tungsten powder with silicon in a reactor with a diameter of 3.8 cm.

The aim of this study is therefore to greatly decrease this weight while maintaining convenient fluidization and thermal conditions to obtain uniform coating characteristics in terms of thickness, chemical composition and morphology. Preliminary experiments have shown that a simple decrease in the bed weight is ineffective because the target deposition temperature cannot be reached due to a too low heat exchange between the reactor walls and the particles. This means that a reduction in the reactor diameter must be considered.

In the literature, the impact of reducing the reactor diameter on gas–solid fluidized bed hydrodynamics has only been studied for powders belonging to Geldart's groups A and B [5], which are easily fluidizable [6–13]. To the best of our knowledge, all studies have been performed at ambient temperature. The early experimental study by Werther [13] has shown that the bed diameter influences bubble development and the distribution of the fluidizing gas between the dense phase and the bubble phase when the bed diameter is smaller than 500 mm. More recently, fluidized beds with inner diameters ranging between 0.7 and 32 mm – called micro-fluidized beds – have been tested for fixed bed heights with reactor diameter ratios (H_0/D) between 0.6 and 7 [6–12]. Below a critical column diameter, there is evidence of changes in the fluidized bed hydrodynamics, known as wall effects. The most significant wall effects are an increase in the minimum fluidization velocity U_{mf}, an increase in the minimum bubbling velocity U_{mb}, and a modification in the bed voidage. Some articles also report a deviation in the experimental pressure drops from the theoretical ones calculated by the Ergun equation, the presence of hysteresis between the ascending and descending pressure drop curves, and overpressure near the minimum fluidization zone [8,12]. The authors found that wall effects were significant for critical column diameters between 5 and 12 mm depending on the conditions tested. Wang et al. [11] have also shown by CFD simulations that the onset of turbulent fluidization is advanced significantly in micro fluidized beds. Wang and Fan [12] have established

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a flow regime map from experimental results obtained in column diameters starting from 0.7 mm, thus confirming this trend.

In a fluidized bed, electrostatic charges can be generated through frictional charging when particles come into contact with other particles or with the column wall. Some significant consequences of charge generation are particle agglomeration and particle build-up on the reactor's inner wall [14]. The existence of electrostatic charges between the reactor walls and the particles (depending on the nature of the wall and of particles) can increase wall effects, especially in micro 2D columns [7]. For Liu et al. [9], the H_0/D ratio has no influence, whereas this ratio increases wall effects for other authors [8,10]. Delebarre [15] and Roche et al. [16] showed that the interaction forces between particles and walls increase with the particle diameter or density. The non-sphericity of particles is also known to increase solid frictions [17]. Liu et al. [18] showed that non-spherical particles can intensify the hysteresis phenomenon between pressure drop curves at ascending and descending gas velocities.

This study analyzes the impact of reducing the column diameter on the fluidization of a very dense tungsten powder under pure argon, first at ambient temperature and then at 650 °C. The aim is to reduce the weight of the surrogate tungsten powder to below 1500 g before reducing the weight of U(Mo) treated by FB-CVD, while maintaining convenient hydrodynamic and thermal conditions in order to produce uniform silicon coatings.

2. Experimental

2.1. Experimental setup and materials

In our experiment, we used glass and steel columns that were 1 m high but with different inner diameters: 5, 3.2 and 2 cm for the glass columns and 5, 3 and 2 cm for the steel columns. A photograph of the 2 cm glass column and a schematic diagram of the steel fluidized bed system are given in Fig. 1a and b. All were equipped with a similar Inox™ porous plate distributor. A differential fast response pressure sensor (GE Druck LPX) with taps under the distributor and on top of the column was used to measure the total pressure drop across the bed. Its accuracy was ±0.5% full scale.

The steel columns were also equipped with a two-zone electrical furnace to simulate FB-CVD conditions with a target bed temperature of 650 °C on average. Five thermocouples were placed into a vertical 6 mm-diameter stainless steel tube inside the reactor to measure the bed temperature within the fluidized bed with an uncertainty estimated at ± 2.5 °C. They were respectively located at 1, 2.5, 5, 7 and 12 cm above the distributor.

All measurements were registered online with a Dasy Lab® acquisition system using an acquisition frequency of 0.1 Hz and a data resolution of 10^{-6}. The fluidization gas was argon (Alpha 1, Air Liquide). Its flow rate was controlled by a mass flow controller (Aera FC-7710 CO, 0–20 slm). All experiments were conducted at atmospheric pressure.

We used a tungsten powder (CERAC, Inc. T-1220) supplied by Neyco. Its grain density was equal to 19300 kg/m^3. Laser grain-size analyses were performed with a Malvern Master Sizer Sirocco 2000 in dry mode. The particle size distribution of the tungsten powder is given in Fig. 2a. The Sauter diameter ($D_{3,2}$) was 70 μm and the D_{10}/D_{90} diameters were equal to 50/105 μm respectively. Fig. 2b shows a SEM photograph of the particles (Philips XL30 FEG) which are clearly non-spherical and faceted.

2.2. Experimental procedure

The fluidization hydrodynamics was first studied by plotting the bed pressure drop versus increasing and decreasing superficial gas velocity in the glass and steel columns at room temperature. Experiments were repeated three times. The distributor pressure drop was first measured within the appropriate range of superficial gas velocities in each empty column and then removed from the experimental pressure drop. Bed pressure drops were averaged over 10 s periods when significant fluctuations occurred. The average expanded bed height was measured visually in the glass columns at decreasing gas velocity. A dimensionless bed expansion H^* was calculated by dividing the experimental expanded bed height by the fixed bed height. A minimum bubbling velocity, U_{mb}, was estimated by visually catching the first bubbles that appeared on the surface of the bed [9]. For each superficial gas velocity, the experiments were run 60 s to measure stationary fluidization data. The minimum fluidization velocity U_{mf} at room temperature was then deduced either from the pressure drop curves using the Davidson and Harrison method [19], or from the bed expansion results [8].

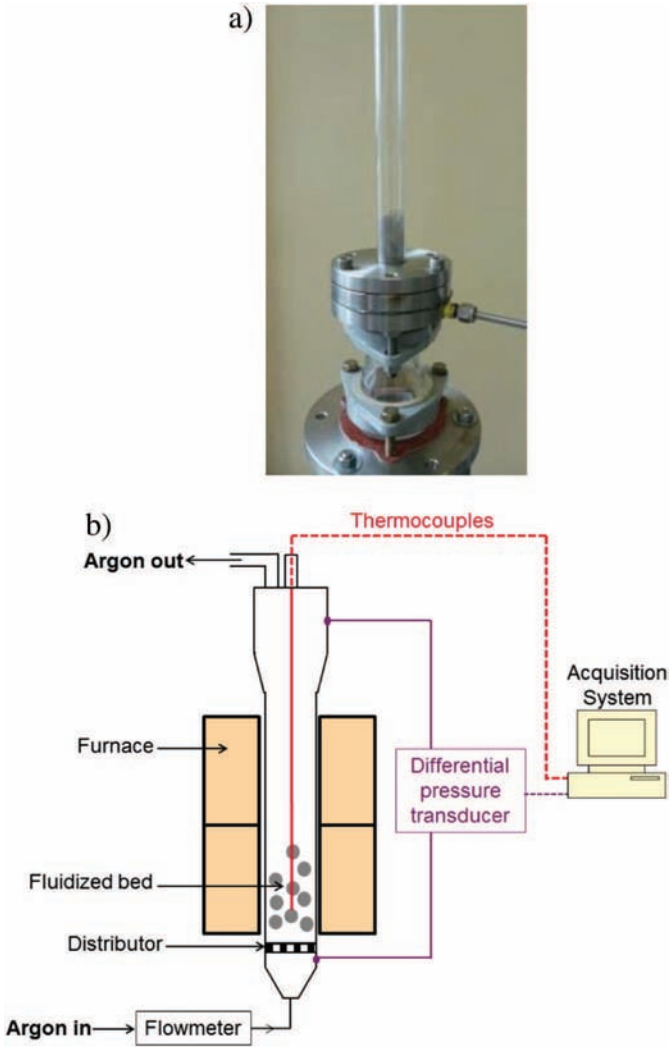

Fig. 1. (a) Photography of the 2 cm glass column – (b) schematic diagram of the steel fluidized bed system.

Experiments were performed using two H_0/D ratios (1.6 and 3) for each column diameter and column wall type (glass or steel). The corresponding powder weights used in each case are detailed in Table 1. A H_0/D ratio close to 3 had already been used for silicon deposition experiments on tungsten particles by FB-CVD using a reactor diameter of 3.8 cm, corresponding to a powder weight of 1500 g.

The second step involved studying the impact of reducing the reactor diameter on the fluidized bed thermal profiles. The aim was to obtain an average bed temperature of 650 °C with a minimal bed thermal gradient in steady-state conditions. The H_0/D ratios and weights of powder tested in the steel reactors are detailed in Table 2. The superficial velocity of argon was fixed around 4 U_{mf}.

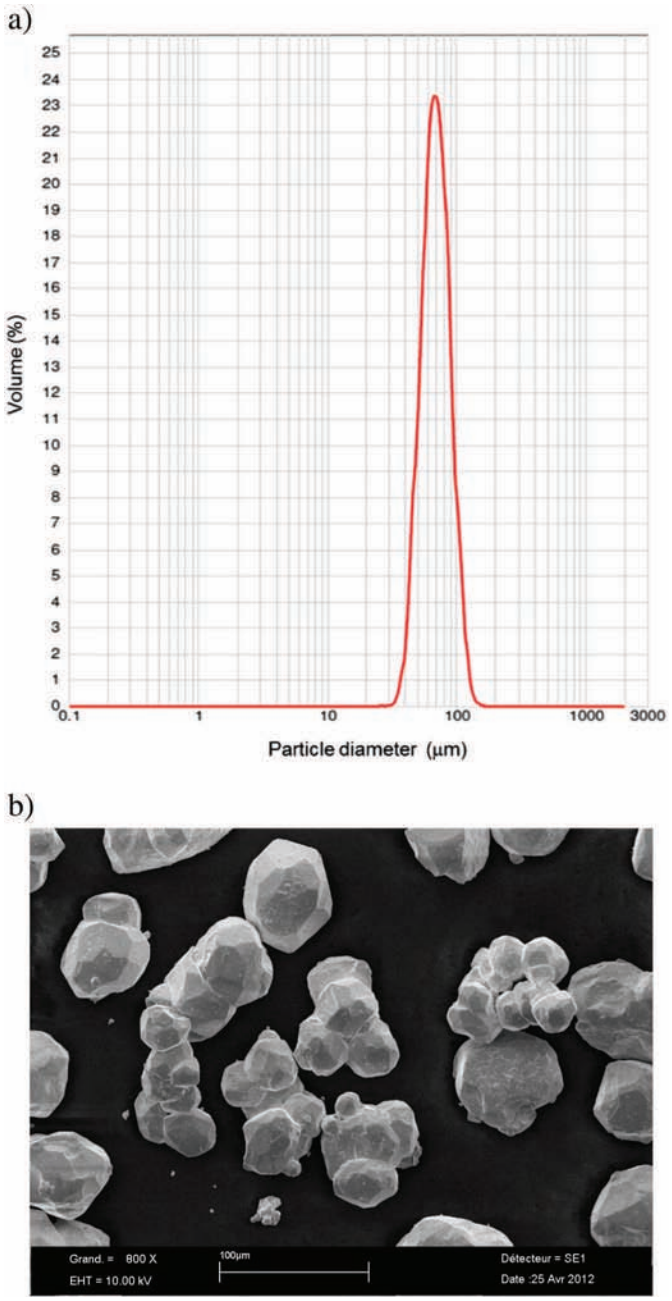

Fig. 2. (a) Particle size distribution of the tungsten powder – (b) SEM view of the tungsten particles.

3. Results and discussion

3.1. Fluidization study at ambient temperature

3.1.1. Determining U_{mf} with the Davidson and Harrison method

Fig. 3 shows the curves of the bed pressure drops which were measured experimentally in the glass and steel columns for the two H_0/D ratios tested at ambient temperature. The error bars include uncertainties due to variations in the ΔP for a given gas velocity, as well as uncertainties due to the pressure sensor (±1.8%) and the argon mass flowmeter (±0.1 cm/s). A horizontal line has been plotted for each experiment, corresponding to the theoretical bed pressure drop (equal to the bed weight per column surface area).

Important information can be deduced from these results. First, a hysteresis phenomenon occurs between the curves at increasing and decreasing argon velocities, which clearly increases when the column diameter decreases regardless of the H_0/D ratio and the type of column walls, though this phenomenon is more pronounced in the glass columns. Such hysteretic behavior is indicative of path-dependent stress into the bed and of non-negligible inter-particle forces [20,21] probably due to the high powder density. Other authors like Srivastava and Sundaresan [22] and Loezos et al. [23] have also observed that the pressure drop hysteresis becomes more pronounced as the size of the fluidized bed is decreased.

Overpressure can also be observed for gas velocities close to the minimum level of fluidization appearing for the 2 cm columns. According to Loezos et al. [23], wall friction can lead to pressure drop overshoots, even for a non-cohesive material.

The experimental pressure drops of the horizontal plateau can be seen to be lower than the theoretical pressure drop for the 2 cm glass columns. We observed that some of the powder remained stuck on the column glass walls due to electrostatic forces, which could explain the lower pressure drops obtained.

Fig. 4 shows the changes in U_{mf} determined by the Davidson and Harrison method from the results of Fig. 3, as a function of the column diameter. U_{mf} remains close to 3 cm/s for the 5 cm and 3 cm column diameters regardless of the column walls and the H_0/D ratio tested, but it increases sharply in the 2 cm column diameters. Moreover, U_{mf} is significantly higher in the 2 cm glass column (3.6–4.1 cm/s) than in the 2 cm steel column (3.2–3.7 cm/s). Wall effects undoubtedly exist in the 2 cm columns and are more pronounced in the glass columns than in the steel ones. The influence of the H_0/D ratio is not significant for these results, as found by Liu et al. [9].

The error bars in Fig. 4 include the uncertainties previously listed and those induced from using the graphical method by Davidson and Harrison. These bars are seen to increase when the reactor diameter decreases and are very significant for the 2 cm column diameter. This can be explained by the fact that the transition zone of the pressure drop curves between the fixed and the fluidized states is less defined for this diameter. This means that the fluidization of the tungsten particles is more difficult.

To better understand our results compared with those of the literature, Table 3 summarizes the conditions we tested and those of the literature, together with the obtained critical column diameter. All the authors worked with powders from Geldart's groups A and B and they all found that the critical column diameters for which the wall effects appeared were significantly lower than ours.

Ranges of column diameters and H_0/D ratios similar to ours have been studied by these authors. Guo et al. [8] used powders of median diameter similar to the tungsten powder. The fluidization gases were different, but as found by Guo et al. [8], this parameter has only a slight influence on the fluidization behavior in columns of reduced diameter. The main difference between our conditions and those of the literature clearly concerns the powder density, which was seven to ten times higher in our case. The hysteretic behavior of the tungsten powder and its low bed expansion mean that its ability to fluidize is lower

Table 1
H_0/D ratios and corresponding weights of the powder tested at ambient temperature.

Column diameter (cm)	5	3 or 3.2	2
$H_0/D \approx 1.6$	1500 g	395 g	97 g
$H_0/D \approx 3$	2827 g	741 g	181 g

Table 2
H_0/D ratios and corresponding weights of the powder in the steel columns at 650 °C.

Reactor diameter (cm)	H_0/D	Weight of powder (g)
5	1.6	1500
3.8	3	1500
3	3	741

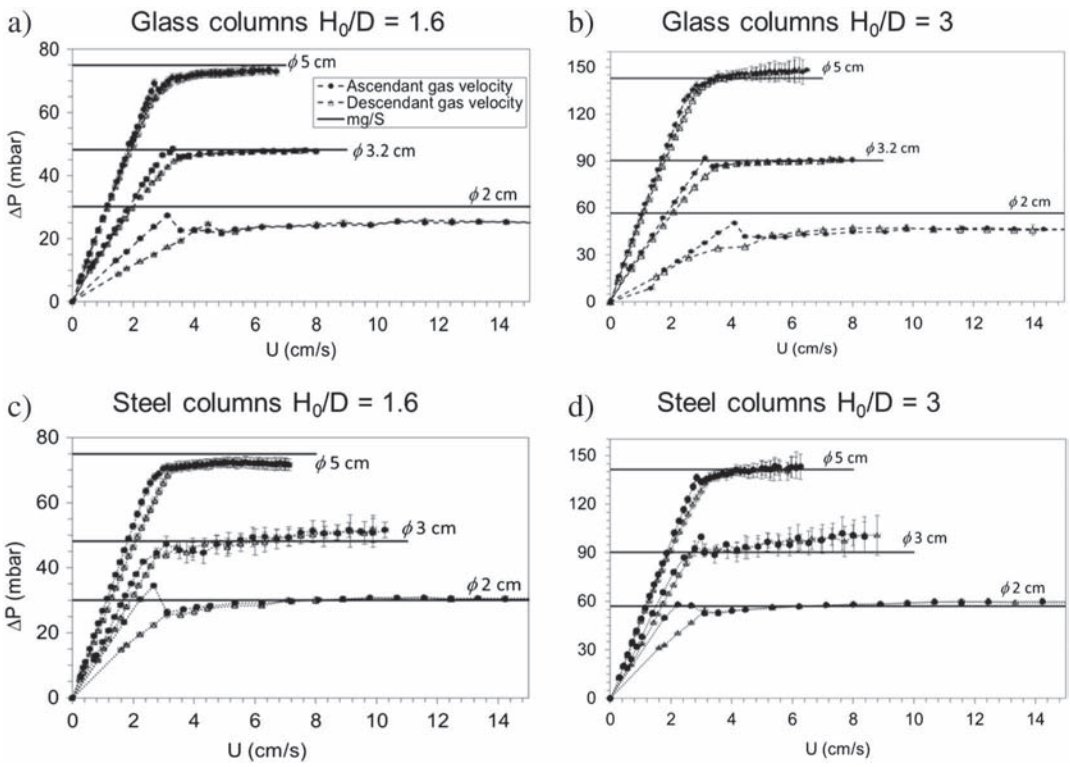

Fig. 3. Experimental bed pressure drop curves in the glass columns for a) $H_0/D = 1.6$ and b) $H_0/D = 3$ and in the steel columns for c) $H_0/D = 1.6$ and d) $H_0/D = 3$.

than that of the powders tested from Geldart's group A and B. Consequently, inter-particle forces and wall effects are more intense for this powder, which explains the higher critical column diameter that we found.

On the basis of their experimental results, Guo et al. proposed a specific empirical correlation to calculate U_{mf} for a micro-fluidized bed. We tested this correlation and some other well-known empirical correlations for the tungsten powder, as detailed in Table 4.

The relations by Wen and Yu [24] and by Todes and Goroskhov [25] obtained a U_{mf} close to 3 cm/s. This value agrees well with the experimental values we obtained in the 5 and 3 cm columns. As found by Guo et al. [8], these correlations are not able to evaluate U_{mf} in columns of reduced diameters since they do not consider the possible impact of wall effects due to a decrease in the column diameter. Yet when using the Guo et al. [8] correlation for our 2 cm column diameter, the calculated U_{mf} values for the two H_0/D ratios tested were much higher than our experimental values. This means that this correlation is probably only valid for Geldart's group A particles. The relative weight assigned to the powder density in this equation is probably too high when applied to the tungsten powder.

3.1.2. Determining U_{mf} from bed expansion measurements

Fig. 5 shows the variation in the dimensionless bed expansion H^* versus the superficial velocity of argon in the glass columns for $H_0/D = 1.6$ and 3 at a decreasing gas flow rate. The error bars mainly correspond to the bed height fluctuations and also include uncertainties due to their visual determination. This absolute uncertainty was estimated at 2 mm for the fixed bed because the bed surface is never perfectly plane, and between 4 and 6 mm for the fluidized bed. Therefore, the corresponding relative uncertainty appears proportionately higher for low bed heights. Furthermore, as previously found [4], the tungsten powder induces low bed expansion (<20%) due to its very high density.

The dimensionless expansion tends to be lower when the column diameter is reduced. This means that the bed voidage decreases significantly with the decrease in the bed diameter due to wall effects. There is no clear consensus in the literature about the impact of reducing the column diameter on the voidage of gas solid fluidized beds with diameters below 50 mm. For Guo et al. [8], the bed voidage increases when the column diameter decreases, whereas for Wang and Fan [12], it is the opposite for quite similar Geldart group A powders but in a lower range of bed diameters.

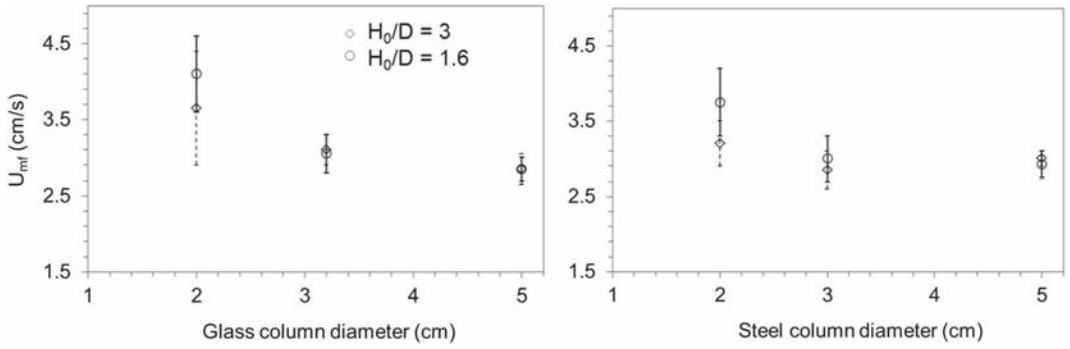

Fig. 4. Variation in U_{mf} versus the column diameter.

Table 3
Comparison of our operating conditions and results with those of the literature.

Authors	Column diameter (mm)	Median diameter of the powder (microns)	Density of the powder (kg/m^3)	H_0/D	Fluidization gas	Critical diameter (mm)
Liu et al.	12 to 32	96 to 460	2600	0.6 to 4.2	Air	12
Guo et al.	4.3 to 25.5	30 to 83	1807 and 2650	1.3 to 3.5	N_2, Air, CO_2	5.5
Sanchez Delgado et al.	5 to 20 (2D beds)	345 to 678	2500	0.5 to 6	–	10
Rao et al.	16 and 24	100 to 550	1250 and 2500	1 to 7	Air	16
Wang and Fan	0.7 to 5	53	1400	–	Air	–
Our study	20 to 50	75	19300	1.6 and 3	Ar	20

When comparing our work with the various studies in the literature (Table 4), as previously explained, the main difference clearly concerns the powder density. In a fluidized bed, particles are submitted to three different major forces, the gravitational force (i.e. the weight of particles), the gas–particle interaction drag force and the particle–particle or particle–wall interaction force. According to Wang et al. [26], when very dense particles are fluidized, the gravitational force dominates the others, leading to a high effective viscosity of the bed. As a consequence, bed expansion is low which in turn means that the number and size of gas bubbles in the bed are low too. When the column diameter is reduced, the ratio of wall contact to bulk volume particles increases, which leads to an increase in the particle wall interaction force [27] and then in the effective viscosity of the bed. This explains the wall effects observed and could also explain the decreased number and size of gas bubbles, as indicated by the bed expansion results.

For the highest column diameters tested, there is a range of gas velocities between the fixed and the bubbling regions where the bed expands homogeneously, i.e. without bubbles. The fact that the minimum bubbling velocity U_{mb} is higher than the minimum fluidization velocity U_{mf} indicates that the tungsten powder presents characteristics of Geldart's group A particles, probably due to its quite low median diameter [5]. However, the difference between these two velocities decreases as the column diameter is reduced. In the 2 cm column diameter, U_{mb} is very similar or equal to U_{mf}. This could be explained by the lower presence of gas in the bed as previously shown, due to wall effects.

The minimum fluidization velocity was classically estimated from Fig. 5 results by considering the intersection of the fixed bed horizontal plateau with the bubbling bed linear region. Fig. 6 shows the variation in U_{mf} with the column diameter for the two H_0/D ratios tested. U_{mf} values similar to those deduced from the pressure drop results were obtained: U_{mf} remains close to 3 cm/s for the 5 and 3.2 cm column diameters and increases to 3.6–4 cm/s in the 2 cm column diameter. These results thus confirm that wall effects are significant for the 2 cm column diameter. Under other conditions, no clear influence of the H_0/D ratio could be found.

3.2. Effect of decreasing the column diameter on the FB thermal profiles at 650 °C

The first part of the study conducted at ambient temperature shows that using a 2 cm-diameter FB-CVD reactor in which the wall effects are significant for the coating application could decrease gas–solid thermal and mass transfer rates into the bed. This could induce a risk of non-uniform coating or even of particle agglomeration. This is why only column diameters greater than 2 cm have been studied at high temperature.

The main objective of this part of the study was to decrease the tungsten bed weight, while reaching an average bed temperature close to 650 °C with a minimum thermal gradient in the bed and without exceeding 800 °C on the reactor walls so as to not damage the equipment. As detailed in Table 5, three column diameters starting from 5 cm and then decreasing to 3 cm were tested. All the results below correspond to the results measured after 80 min of heating.

The average temperature in the fluidized bed corresponds to the average of the temperatures measured by the 4 thermocouples between 1 and 7 cm above the distributor because the thermocouple at 12 cm was not always inside the fluidized bed. The bed's thermal gradient is the temperature difference between the maximal temperature measured by the thermocouple located at 7 cm, and the minimal temperature in the fluidized bed generally registered by the thermocouple located at 1 cm above the distributor.

For the first experiment in the 5 cm-diameter reactor, a H_0/D ratio of 1.6 was used, corresponding to a bed weight of 1500 g. The average bed temperature did not exceed 362 °C for a wall temperature close to 740 °C. In fact, the height of powder in contact with the heated reactor walls was too small to ensure convenient FB heating for this rather large diameter. This is mainly due to the facts that (i) expansion of the tungsten bed is low and (ii) the reactor part close to and below the distributor cannot be heated as it would cause the silane to decompose prematurely, which could lead to distributor clogging.

The experiment was repeated in a smaller reactor diameter, 3.8 cm and a H_0/D ratio of 3, corresponding also to a bed weight of 1500 g. For a wall temperature of 738 °C, the average bed temperature was close to 650 °C and the thermal gradient was only of 2 °C within the fluidized bed. These conditions, which have already been tested for silicon deposition, are very favorable for CVD coating. At this reactor diameter at least, it appears that a H_0/D ratio of 3 guarantees good heat transfer between the reactor walls and powder and then ensures a uniform bed temperature.

In the last stage of this study, the H_0/D ratio of 3 was then maintained and the reactor diameter was decreased to 3 cm in order to reduce the powder weight to 740 g. An average bed temperature close to 650 °C was also reached, but the reactor walls had to be heated to 780 °C and the bed's thermal gradient increased to 15 °C. This means that the bed hydrodynamics and the resulting thermal transfer between the powder and the reactor walls were of lower quality than in the reactor with a 3.8 cm diameter. Therefore, even if the minimum fluidization velocity measured at ambient temperature remains unchanged

Table 4
Empirical equations of the literature and calculated U_{mf} values for the tungsten powder.

Authors	Range of applicability	Equation	U_{mf} (cm/s)
Wen and Yu (1966)	Not limited	$Re_{mf} = \sqrt{33.7^2 + 0.0408\frac{d_p^3 \rho_g (\rho_p - \rho_g) g}{\mu_g^2}} - 33.7$	3.07
Todes and Goroskhov (1957)	Not limited	$Re_{mf} = \frac{Ar}{1400 + 5.22 Ar^{0.50}}$	3.42
Guo et al. (2009)	Micro-fluidized bed	$U_{mf} = \left[\frac{H_0}{d_p} e^{-6.312 + 242.272/(D/d_p)} + 1\right] \times \frac{7.169 \times 10^{-4} d_p^{1.82} (\rho_p - \rho_g)^{0.94} g}{\rho_g^{0.06} \mu_g^{0.88}}$	7.8 for D = 2 cm and H_0/D = 1.6 11.9 for D = 2 cm and H_0/D = 3

$Re_{mf} = \frac{d_p U_{mf} \rho_g}{\mu_g}$, $Ar = g d_p^3 \rho_g \frac{(\rho_p - \rho_g)}{\mu_g^2}$.

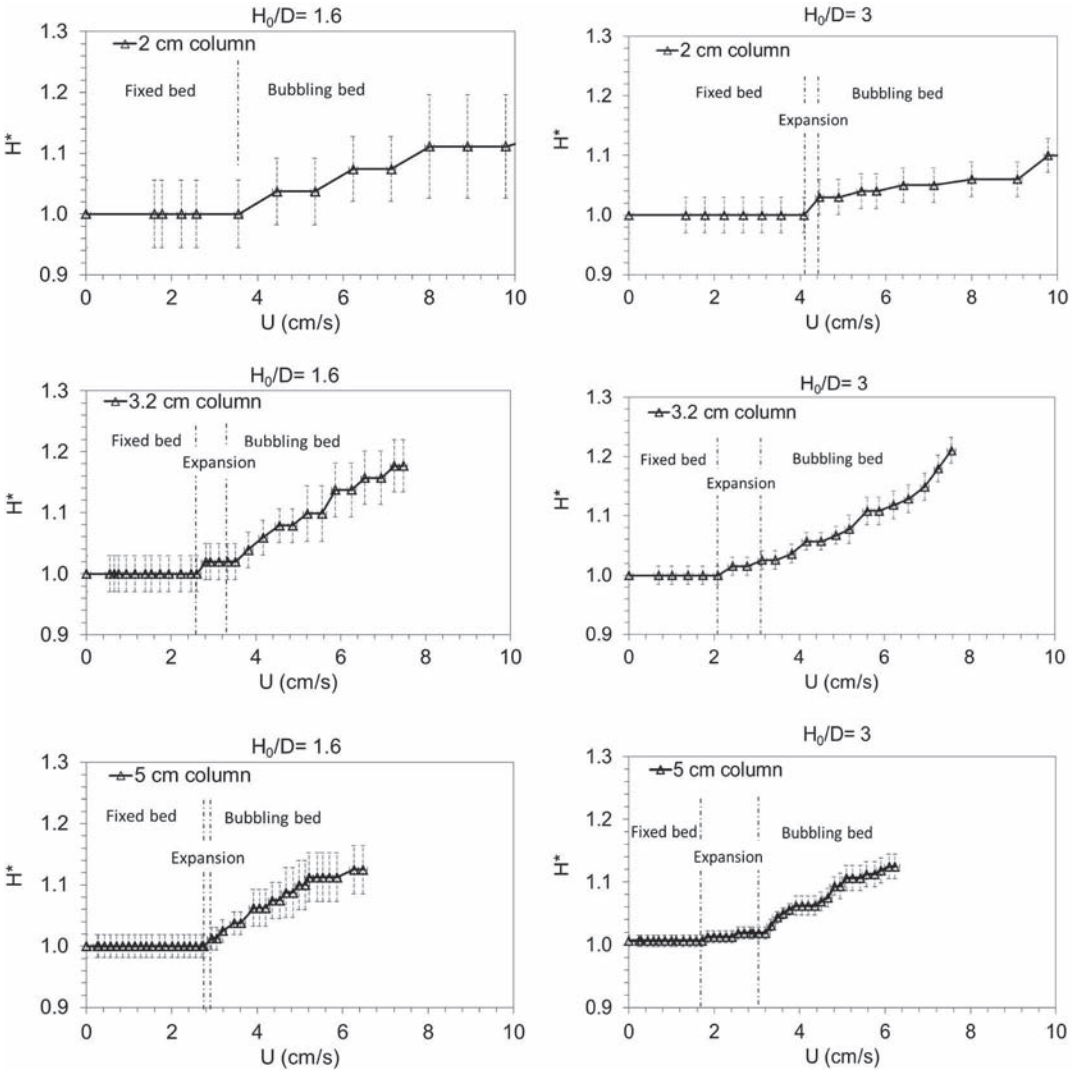

Fig. 5. Variation in the bed dimensionless height versus argon superficial velocity in the glass columns for the two H_0/D ratios tested.

between column diameters of 5 and 3 cm, it seems that the wall effects already exist in the 3 cm column diameters, which are much less intense than in the 2 cm ones. Consequently, the thermal behavior of the FB seems to be more sensitive to a decrease in the reactor diameter than the pressure drop or expansion measurements at ambient temperature.

According to previous CVD process results obtained within our group, Rodriguez et al. [4] showed that a thermal gradient of 40 °C was acceptable to obtain a uniform CVD coating. For this reason, the next FB-CVD experiments with silicon coating will be conducted using a tungsten bed of 740 g in a column with a 3 cm diameter.

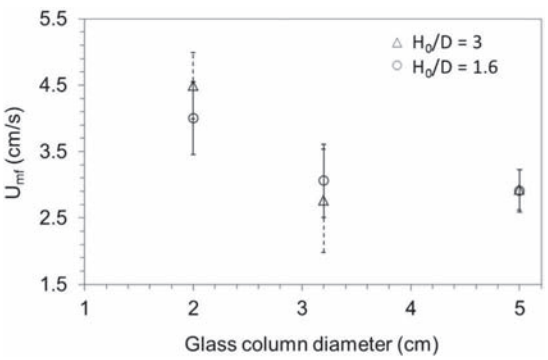

Fig. 6. Variation in U_{mf} versus the glass column diameter for the two H_0/D ratios tested.

4. Conclusion

This paper has investigated the impact of decreasing the column diameter on the fluidization of a very dense tungsten powder. Pressure drop and expansion measurements were first performed at ambient temperature in glass and steel columns with inner diameters between 2 and 5 cm for H_0/D ratios of 1.6 and 3. The wall effects were evidenced for the 2 cm column diameter, including an increase in the hysteretic behavior between increasing and decreasing pressure drop curves, an increase in the minimum fluidization velocity and a decrease in bed voidage. This critical diameter is higher than those found in other studies of the literature for powders from Geldart's groups A and B, certainly due to the very high density of the tungsten powder which enhances the friction forces in the bed.

Fluidized bed thermal profiles at about 650 °C were measured for reactor diameters between 3 and 5 cm and H_0/D ratios of 1.6 and 3, in order to find conditions making it possible to decrease the bed weight below 1500 g while maintaining minimal bed thermal gradients. Suitable conditions were found in the 3 cm-diameter reactor with an

Table 5
Variation in the average wall and bed temperatures and bed thermal gradients for various reactor diameters and H_0/D ratios.

Reactor diameter (cm)	Bed weight (g)	H_0/D	Temperature of reactor walls (°C)	Average temperature in the fluidized bed (°C)	Thermal gradient in the bed (°C)
5	1500	1.6	740	362	6
3.8	1500	3	738	637	2
3	740	3	780	640	15

H_0/D ratio of 3, corresponding to 740 g of powder. This therefore opens the way to reducing the powder weight during future FB-CVD experiments of tungsten powder coating using silicon.

Notation

Latin letters		
Ar	(−)	Archimedes number
d_p	(m)	Median diameter of particles
D	(m)	Reactor diameter
g	(m/s^2)	Gravitational acceleration
H	(m)	Fluidized bed height
H_0	(m)	Fixed bed height
H^*	(−)	Ratio of the fluidized bed height by the fixed bed height
Re_{mf}	(−)	Particular Reynolds number at the minimum of fluidization
U	(m/s)	Superficial gas velocity
U_{mb}	(m/s)	Minimum bubbling velocity
U_{mf}	(m/s)	Minimum fluidization velocity
Greek letters		
ΔP	(Pa)	Bed pressure drop
μ_g	(Pa.s)	Gas viscosity
ρ_g	(kg/m^3)	Gas density
ρ_p	(kg/m^3)	Particle density

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